



PST/GB 2003 / 0 0 3 4 3 4



INVESTOR IN PEOPLE

The Patent Office
Concept House
Cardiff Road
Newport

South Wales NP10 8QQ

WIPO PCT

PRIORITY DOCUMENT

SUBMITTED OR TRANSMITTED IN
COMPLIANCE WITH RULE 17.1(a) OR (b)

I, the undersigned, being an officer duly authorised in accordance with Section 74(1) and (4) of the Deregulation & Contracting Out Act 1994, to sign and issue certificates on behalf of the Comptroller-General, hereby certify that annexed hereto is a true copy of the documents as originally filed in connection with the patent application identified therein.

In accordance with the Patents (Companies Re-registration) Rules 1982, if a company named in this certificate and any accompanying documents has re-registered under the Companies Act 1980 with the same name as that with which it was registered immediately before re-registration save for the substitution as, or inclusion as, the last part of the name of the words "public limited company" or their equivalents in Welsh, references to the name of the company in this certificate and any accompanying documents shall be treated as references to the name with which it is so re-registered.

In accordance with the rules, the words "public limited company" may be replaced by p.l.c., P.L.C. or PLC.

Re-registration under the Companies Act does not constitute a new legal entity but merely subjects the company to certain additional company law rules.

Signed

Dated

18 August 2003

Best Available Copy

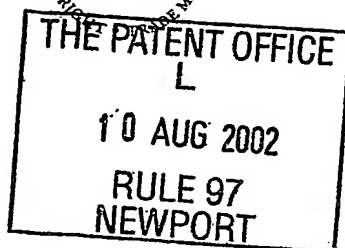


12AUG02 1740297-1 D02827
P01/7700-0.00-0218672.4

1/77

Request for grant of a patent

(See the notes on the back of this form. You can also get an explanatory leaflet from the Patent Office to help you fill in this form)



The Patent Office

Cardiff Road
Newport
South Wales
NP10 8QQ

1. Your reference 4636

2. Patent number 0218672.4

10 AUG 2002

3. Full name, address and postcode of the or of each applicant (underline all surnames)

Pilkington plc
Prescot Road
St Helens
Merseyside
WA10 3TT

Patents ADP number (if you know it)

If the applicant is a corporate body, give the country/state of its incorporation

England

660449001

4. Title of the invention FIRE RESISTANT GLAZINGS

5. Name of your agent (if you have one)

Anthony Charles Halliwell

"Address for service" in the United Kingdom to which all correspondence should be sent (including the postcode)

Group Intellectual Property
Pilkington plc
European Technical Centre
Hall Lane,
Lathom, Ormskirk
Lancashire L40 5UF

Patents ADP number (if you know it)

7207897001

6. If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and (if you know it) the or each application number

Country	Priority application number (if you know it)	Date of filing (day / month / year)
-	-	-

7. If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application

Number of earlier application	Date of filing (day / month / year)
-	-

8. Is a statement of inventorship and of right to grant of a patent required in support of this request? (Answer 'Yes' if:

- a) any applicant named in part 3 is not an inventor, or
 - b) there is an inventor who is not named as an applicant, or
 - c) any named applicant is a corporate body.
- See note (d))

YES

Patents Form 1/77

Enter the number of sheets for any of the following items you are filing with this form. Do not count copies of the same document

Continuation sheets of this form

Description

7

Claim(s)

3

Abstract

1

Drawing(s)

0

10. If you are also filing any of the following, state how many against each item.

Priority documents

-

Translations of priority documents

-

Statement of inventorship and right to grant of a patent (Patents Form 7/77)

-

Request for preliminary examination and search (Patents Form 9/77)

1

Request for substantive examination (Patents Form 10/77)

-

Any other documents (please specify)

-

11.

I/We request the grant of a patent on the basis of this application.

Signature

R Hamilton

Date 09/08/02

R Hamilton in the absence of Anthony Charles Halliwell (Agent for the Applicant)

12. Name and daytime telephone number of person to contact in the United Kingdom

R Hamilton - 01695 54354

Warning

After an application for a patent has been filed, the Comptroller of the Patent Office will consider whether publication or communication of the invention should be prohibited or restricted under Section 22 of the Patents Act 1977. You will be informed if it is necessary to prohibit or restrict your invention in this way. Furthermore, if you live in the United Kingdom, Section 23 of the Patents Act 1977 stops you from applying for a patent abroad without first getting written permission from the Patent Office unless an application has been filed at least 6 weeks beforehand in the United Kingdom for a patent for the same invention and either no direction prohibiting publication or communication has been given, or any such direction has been revoked.

Notes

- If you need help to fill in this form or you have any questions, please contact the Patent Office on 08459 500505.
- Write your answers in capital letters using black ink or you may type them.
- If there is not enough space for all the relevant details on any part of this form, please continue on a separate sheet of paper and write "see continuation sheet" in the relevant part(s). Any continuation sheet should be attached to this form.
- If you have answered 'Yes' Patents Form 7/77 will need to be filed.
- Once you have filled in the form you must remember to sign and date it.
- For details of the fee and ways to pay please contact the Patent Office.

Best Available Copy

Patents Form 1/77

FIRE RESISTANT GLAZING

This invention relates to novel solutions useful in the production of fire resistant glazings comprising an alkali metal aluminate and a silicate waterglass, to methods for the preparation of those solutions and to the production of intumescent interlayers from those solutions which may be incorporated into fire resistant glazings..

Glass laminates incorporating an intumescent inorganic silicate layer sandwiched between two opposed panes of glass are sold under the trade marks PYROSTOP and PYRODUR by the Pilkington group of companies. When such laminates are exposed to a fire the inorganic layer intumesces and expands to form a foam. The foam provides a thermally insulating layer which protects the pane of glass remote from the fire so that the structural integrity of the glass unit and thereby a barrier to the propagation of the fire is maintained for a longer period. Glass laminates incorporating such intumescent layers have been used successfully as fire resistant glass structures. These laminates may comprise more than two panes of glass sandwiching more than one intumescent interlayer. Laminates comprising up to eight intumescent layers have been employed. These multi layered laminates are relatively thick and correspondingly expensive.

The intumescent inorganic layer is normally formed from a sodium silicate waterglass or a mixture thereof with a potassium silicate waterglass. The layer is commonly formed by preparing a solution of the waterglass (or waterglasses), spreading that solution on the surface of the glass and drying excess water from the solution so as to form the intumescent inorganic layer.

USP 4190698 discloses fire resistant glazings comprising an intumescent inorganic layer obtained by drying a waterglass solution. The authors suggest the addition of various additives to the waterglass solution including urea, polyhydric alcohols, monosaccharides, polysaccharides, sodium phosphate, sodium aluminate, borax, boric acid and colloidal silica. There is no specific disclosure of the addition of an aluminate to a waterglass solution.

We have discovered that the addition of sodium aluminate to a waterglass solution as proposed in USP 4190698 does not produce a solution which is useful in the production of fire resistant glazings. The solutions are unstable and form a precipitate

In particular preferred embodiment the waterglass solution used in the present invention comprise a mixture of sodium silicate waterglass and a potassium silicate waterglass. The molar ratio of sodium ions to potassium ions in these mixtures is preferably at least 4:1. Where a potassium silicate waterglass is employed it is preferably one where the weight ratio $\text{SiO}_2 : \text{K}_2\text{O}$ is in the range 1.43:1 to 2.05:1.

The solutions of this invention may further comprise one or more polyhydric compounds which are known to be useful in existing intumescent interlayers. Polyhydric compounds which have been proposed for this use include glycerol, glycerine or a derivative of glycerine or a sugar. The most commonly used polyhydric compound and the preferred polyhydric compound for present use is glycerol.

In order to be useful in the production of intumescent interlayers the solution of this invention should preferably be clear, stable and capable of being dried to form a useful intumescent interlayer. The properties of the solution are affected by the composition of the solution and the methods used in their preparation. The utility of any particular solution may be determined by experiment. The stability of the solutions decreases as the amount of aluminate present increases. However the fire resistance of the interlayer increases in proportion to the amount of aluminate present. The amount of aluminate which is incorporated into the solution will preferably represent a compromise between these two properties and will typically be such that the molar ratio of silicon to aluminium is in the range 20:1 to 35:1 more preferably in the range 25:1 to 32:1.

A second significant factor which affects the utility of the solutions of this invention is the weight ratio of silica to alkali metal oxide. Increasing the proportion of silica reduces the stability of the solution and is undesirable. The lower ratios of silicon to alkali metal are preferred since this increases the flow point of the dried interlayer which is an important factor influencing the performance of a glazing incorporating such an interlayer in fire testing. Generally we prefer that the weight ratio of silica to alkali metal oxide is in the range 2:1 to 4:1.

The solutions of the present invention must be clear. They are prepared by mixing the various components in a manner which produces a clear stable solution which may then be dried to form a clear stable intumescent layer.

We prefer that the solutions are prepared using a process which comprises as a first step partially neutralising the aluminate with the hydroxy carboxylic acid. This

The solution is then dried under carefully controlled conditions of temperature and humidity so as to ensure the production of a clear transparent interlayer which is free from bubbles and other optical imperfections. The dried interlayers generally comprise from 10 to 35% by weight of water. The aluminium content of the dried interlayer is generally in the range 0.1% to 5.0% by weight preferably from 0.1% to 1.0% by weight. We have discovered that the presence of the aluminium improves the performance of glazings into which the interlayer is incorporated in terms of their fire resistance and their mechanical impact resistance properties.

The thickness of the dried interlayer will generally be in the range 0.1 to 2.0 mm preferably from 0.5 to 2.0 mm. The formations of thicker interlayers requires a longer drying time and is thereby disadvantageous. Thinner interlayers can be produced using shorter drying times. A glass laminate having a thicker interlayer may be produced by bringing two sheets of glass each having a relatively thin interlayer having say a thickness of from 0.5 mm to 1.0 mm into face to face contact so as to form a laminate having an intumescent interlayer which is from 0.2 to 2.0 mm thick.

Flat glass sheets of various thickness may be used in the laminates of the present invention. Typically sheets of soda lime float glass having a thickness of from 2.0 mm to 4.0 mm are employed.

The edge barrier is normally cut away at the completion of the drying process to leave a glass sheet having a dried interlayer on one surface. A laminate may be formed by placing a second glass sheet on top of the interlayer. In another embodiment the second glass sheet may itself be one having an intumescent interlayer on one surface thereof. Mounting this second sheet on top of the first sheet so that the two fire resistant interlayers are in contact with each other produces a laminate having a relatively thick interlayer. Mounting the second sheet so that the interlayer is on its upper surface and subsequently providing a third glass sheet on top of that second interlayer produces a laminate having two interlayers mounted between three panes of glass. Laminates having as many as eight interlayers may be produced.

In an alternative process the solutions of this invention may be poured onto the surface of the substrate and dried to form an intumescent fire resistant interlayer which is sufficiently strong to be removed from the substrate in the form of a transparent film. The film may then be placed between two sheets of glass to form a fire resistant glass

A waterglass solution comprising a sodium silicate, a potassium silicate and glycerol was made up by mixing 151.7 parts by weight of Crystal 96 with 44.3 parts by weight of Crystal K120 and 20.5 parts by weight of glycerol.

The mixed solution comprising the aluminate was then added to the waterglass solution. The addition was carried out by slowly adding the aluminate solution with thorough mixing using a Silverson high shear mixer. The resulting solution was clear and was stable on storage at room temperature.

This solution was then applied to the surface of a sheet of float glass having an edge barrier around its perimeter in a quantity of 4 kilograms of solution per square metre of glass. The glass sheet was placed in an oven and dried over a prolonged period in a controlled atmosphere until the water content of the solution had reduced to 26% by weight. A clear interlayer having a depth of approximately 1.3mm had formed on the surface of the glass.

The edge barrier was cut away and a second sheet of float glass having a thickness of 3mm was placed on top of the interlayer to produce a glass laminate. Pieces of this laminate were tested for Fire Resistance according to B.S. 476 Part 20/2 and mechanical impact performance according to B.S. 6206 Class C. Two pieces were subject to the fire test and both passed with times of 33 minutes and 30 minutes. Four pieces were tested for impact performance and all were rated at least a safe pass.

- 11 A solution according to either of claims 9 or 10 characterised in that the molar ratio of sodium ions to potassium ions is at least 2:1.
- 12 A solution according to any of the preceding claims characterised in that the molar ratio of silicon to aluminium is in the range 20:1 to 35:1.
- 13 A solution according to claim 12 characterised in that the molar ratio of silicon to aluminium is in the range 25:1 to 32:1.
- 14 A solution according to any of the preceding claims characterised in that the weight ratio of silica to alkali metal oxide is in the range 2:1 to 4:1.
- 15 A solution according to any of the preceding claims characterised in that it further comprises a polyhydric compound.
- 16 A solution according to claim 15 characterised in that the polyhydric compound is glycerol.
- 17 A clear intumescent interlayer characterised in that it has been produced by drying a solution according to any of claims 1 to 16 under controlled conditions.
- 18 An interlayer according to claim 17 characterised in that it comprises from 10 to 35% by weight of water.
- 19 An interlayer according to either of claims 17 or 18 characterised in that it comprises from 0.1 to 5.0% by weight of aluminium.
- 20 An interlayer according to any of claims 17 to 19 characterised in that the interlayer has a thickness of from 0.5 to 2.0 mm.

ABSTRACT

Fire resistant glazings comprise a fire resistant interlayer based upon a silicate waterglass are characterised by the incorporation of aluminium ions. The aluminate is incorporated as a solution which has been partially neutralised using hydroxycarboxylic acid which is preferably citric acid. The incorporation of aluminium provides glazings have improved fire resistance and impact properties